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Fatigue resistance of light alloy sheets undergoing eco-friendly chemical milling: metallurgical and chemical aspects

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Abstract

Component lightening is a key issue for automotive and aerospace industries. Lightening processes on design profile are not always possible by means of traditional machining processes. Then, chemical milling processes are used, often by acid etching. The effect on fatigue behavior is related on many factors, such as chemical surface composition and surface roughness. Material removal through chemical milling is in particular interesting for Additive manufactured components where surface finish still remains a parameter difficult to be controlled and repeated. The environmental aspects related to alkaline and acid processing are still an important issue.
In the present paper, an overview on chemical milling for component lightening is presented with focus of the effects on mechanical and chemical resistance of materials.
Then, the results of an experimental campaign on an aluminum alloy is presented. In particular, high cycle fatigue tests results are presented on specimens subjected or not subjected to an eco-friendly alkaline chemical milling process (called " Green Etching") and chemical, profilometric and metallographic analyses are presented and related to fatigue resistance results. Wettability and surface charge results will be presented.

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Keywords: chemical etching; light alloys; fatigue resistance

1. Introduction

Chemical milling is documented since 2500 B.C., when it was used for copper jewels manufacturing by means of citric acid in Egypt (Harris, 1976). In the 1950s, chemical milling was accepted as one of the most important non-conventional manufacturing techniques. Industrial implementation is due to M. C. Sanz and North Aviation Inc. in

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1956 when it was patented in USA (Sanz, 1956). Applications can be found in aeronautical production (Çakır 2008) and in automotive and rail production. The procedure can be applied to complex geometries, sheets, forged and extruded parts Today, this procedure is used for thin sheet processing of complex components and for weight reduction of components like airplane wings or fuselage (Drozda and Wick (1989), Çakır (2007)).

Aim of this research is to investigate the effect of Green Etching® on a 7075-T6 aluminum alloy, used in automotive and aerospace application, on mechanical properties.

2. Materials and methods

The investigated material is a 7075-T6 aluminum alloy sheet, 4mm thick; its chemical composition and tensile properties are reported In Table 1. Flat tensile and fatigue specimen were designed in compliance of ASTM E466-15 and were manufactured by means of laser cutting (Figure 1). A set of specimens was tested in the as-fabricated condition, while two sets of specimens underwent Green Etching®, one at 50°C for 145 min and the other at 80°C for 26 min. The processing time was chosen in order to obtain a thickness reduction of 1 mm, that is from 4 to 3 mm.

Table 1. Chemical composition of the investigated 7075 alloy (mass fraction %) and mechanical properties (MPa).

Zn	Mg	Cu	Mn	Cr	Si	Fe	Al	UTS [MPa]	σy [MPa]
5,23	2,1	1,45	0,3	0,16	0,23	0,22	Bal	560	480

For microscopic analysis, a Leica Z16 AP0A (115x) optical microscope was used, while, for metallographic analysis, a Reichert-Jung MeF3 (1000x) optical microscope was used. In particular, metallographic specimens were polished and then etched with the Keller solution for 30s.

For surface roughness measurements, an ALPA RTP 80 Metrology System instrument was used. In particular the following roughness parameters were measured: *Ra, Rq, Rt, Rz, Rsk, Rku* and *RSm.* According to Standards definitions they are all parameters related to the amplitude of roughness, i.e. the distances between peaks and valleys. *Ra* is the arithmetical mean deviation of the assessed profile; *Rq* is the root mean square of the mean deviation of the assessed profile, it is an average amplitude measurement in the height direction; *Rt* is maximum height of the profile, that is the distance between the maximum peak and the minimum valley, it is related to the total height of the profile; *Rz* is the average distance between the highest peak and lowest valley in each sampling length, it is related to the maximum height of the profile; two parameters are average characteristics in the height direction, *Rsk* the skewness and *Rku* the kurtosis; *RSm* is the mean width of the profile elements that is a characteristic measured in the surface direction.

For Vickers microhardness measurements, Remet HX1000 tester was used with 10 g load. 5 measures were acquired in the core and 5 in subsurface with about 30 μm distance each. Average measurement on a set of 5 was then calculated.

For uniaxial HCF fatigue testing (room temperature), an Amsler 10 HFP 422 was used (100kN load cell). In particular, 2.10^6 cycles, 125 Hz, 100 MPa mean value, fatigue strength characterization $\sigma_{D\,(50\%)}$ was meant as the identification of the 2.10^6 cycles knee in the SN diagram. It was performed by means of the staircase method (UNI 3964).

For SEM observation, wettability test and zeta potential titration, rectangular samples (10x20 mm) were obtained by cutting, washed one time (5 min) in acetone and subsequently two times (10 min each) in ultrapure water in an ultrasonic bath before analysis. The surface morphology of as received and treated samples was investigated by means of Scanning Electron Microscopy (SEM, JEOL, JCM 6000 plus) and the same samples were subjected to semi-quantitative chemical composition by means of Energy Dispersive Spectroscopy (EDS, JEOL, JED 2300). Surface wettability was investigated by means of contact angle measurements with ultrapure water. 6 measurements were performed for each type of sample. Statistical analysis of the data was performed by means of one-way ANOVA.An electrokinetic analyzer (SurPASS, Anton Paar) has been employed for zeta potential titration vs pH. The surface zeta potential was determined in function of pH in a 0,001 M KCl electrolyte solution varying the solution pH by addition of 0,05M HCl or 0,05M NaOH through the instrument automatic titration unit. The acid and alkaline sides of the curve were obtained in two different steps on a different set of samples.

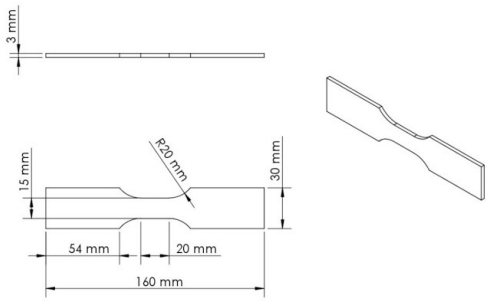


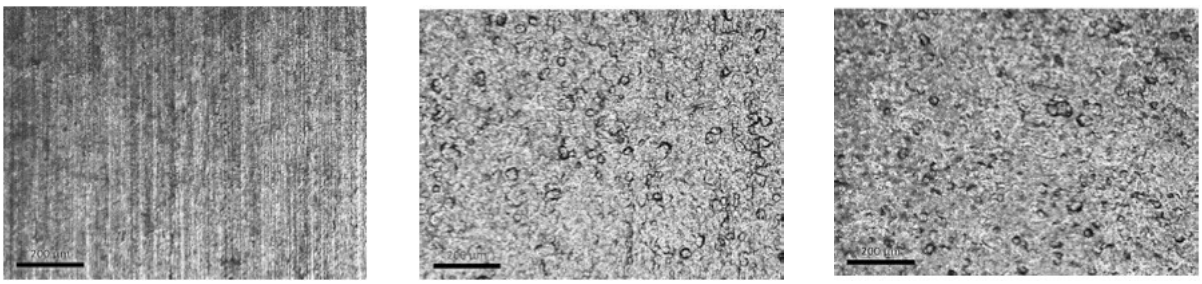
Fig. 1. Specimen design for fatigue testing.

3. Results and discussion

3.1. Microscopic and Metallographic analysis

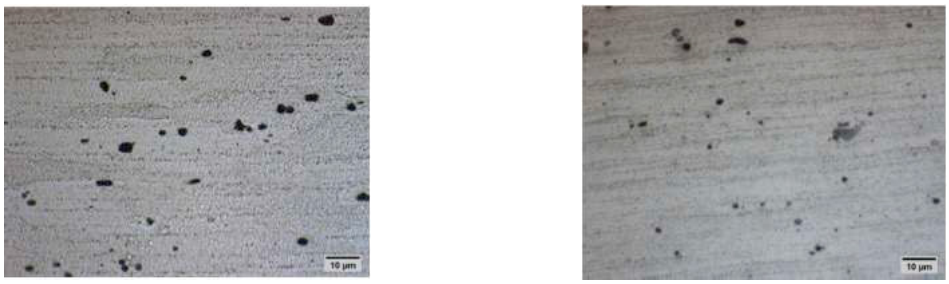
In the following Figure 2 the microscopic analysis of the surface of as-received and GE (green etched) specimens. Figure 2 reports low magnification (115x) images of surface of as received and treated specimens. Figure 2(a) shows the typical traces of lamination process for as received specimen, while in Figures 2(b) and 2(c) these traces have been removed and shallow pits due to chemical processing, with homogeneous distribution, are evident.

In Figure 3, the metallographic analysis on a cross section of two specimens (as received and 80°C treated) is reported. It can be observed that no variation was found in the metallographic structure of treated and as received samples. The investigated surfaces show grain stretching due to lamination process. Both samples show precipitates at the edges of grains with homogeneous distribution in both cases. No differences can be found between internal and external areas for treated and untreated samples. This shows that the Green Etching® process does not modify the superficial metallographic structure of the sample.



(a) *(b)* *(c)*

Fig. 2. Metallographic analysis of the surface of 7075-T6 specimens: (a) as received; (b) 50°C treated; (c) 80°C treated.



(a) *(b)*

Fig. 3. Metallographic analysis of the cross section of 7075-T6 specimens: (a) as received; (b) 80°C treated.

3.2. Surface roughness

The following parameters were measured on the specimens: *Ra, Rq, Rt, Rz, Rsk, Rku* and *RSm*. Measuring parameters are: cutoff 0.8 mm, measuring length 50 mm, speed 0.5 mm/s. Four measurements were done on each specimen and 10 specimens per set were measured. For each set and for each parameter, the average value was calculated. Measurements were acquired in both rolling and transversal direction. In Table 2 to 4, the results are reported for as received, 50°C treated and 80°C treated specimens, respectively.

Table 2. surface roughness measurements. As received specimens, average values (μm).

		Ra	*Rq*	*Rt*	*Rz*	*Rsk*	*Rku*	*RSm*
Rolling Direction	Average	0.108	0.137	1.023	0.719	-0.085	3.132	57.900
	Standard deviation	0.019	0.024	0.248	0.124	0.111	0.423	9.514
Transversal Direction	Average	0.208	0.262	1.693	1.324	-0.241	3.323	41.400
	Standard deviation	0.033	0.044	0.529	0.225	0.156	0.871	3.119

Table 3. surface roughness measurements. 50°C Green Etching treated specimens, average values (μm).

		Ra	*Rq*	*Rt*	*Rz*	*Rsk*	*Rku*	*RSm*
Rolling Direction	Average	1.370	1.726	11.101	8.505	-0.043	3.136	91.450
	Standard deviation	0.132	0.169	1.971	0.906	0.153	0.377	7.373
Transversal Direction	Average	1.536	1.921	11.922	9.432	-0.116	3.118	96.100
	Standard deviation	0.133	0.151	1.249	0.754	0.093	0.356	12.756

Table 4. surface roughness measurements. 80°C Green Etching treated specimens, average values (μm).

		Ra	*Rq*	*Rt*	*Rz*	*Rsk*	*Rku*	*RSm*
Rolling Direction	Average	1.222	1.531	9.264	7.341	-0.093	2.943	100.400
	Standard deviation	0.083	0.107	1.077	0.586	0.093	0.318	12.420
Transversal Direction	Average	1.375	1.737	11.176	8.610	-0.111	3.132	97.900
	Standard deviation	0.101	0.132	1.642	0.881	0.104	0.242	16.717

Higher values are generally measured in the transversal direction and higher values of all parameters are measured for treated specimens. *Ra, Rq* and *Rt* values show increased values after processing and, especially for lower treatment temperature, corresponding to longer treatment duration. In Peter et al. (2019), the material removal efficacy is shown to be more effective with increasing temperature, while in the present research it is shown that the surface roughness results to be more influenced by the process time. *Rz* shows a different trend. It increases with process temperature and decreases with process time. This can be due to the fact that, as *Rz* is related to the maximum distance between the higher peek and the lower valley in the measuring length, the GE process tends to smooth the peaks. *Rsk, Rku, RSm* are related to shape and dimension of profile peaks and valleys. *Rku* tends to 3 with increasing temperature. GE process smooths the profile by increasing the curvature radius of the peaks and of the valleys. *RSm* increases with processing temperature this meaning that valleys are wider and more symmetrical (UNI EN ISO 4287 (2011)).

3.3. Micro hardness (10g)

Measurements were performed on 5 specimens per each sample. Two areas were measured on the cross section: close to the surface and in the core. In Table 5, the average measurements are reported for as received and 80°C treated specimens.

Table 5. microhardness average measurements.

	surface	core
As received	110,2	92,0
80°C treated	111,8	98,1

(a) (b)

Microhardness increases from core to surface and the increment is lower for processed specimens. For as received specimens, the lamination process is responsible for higher surface hardness. It seems that GE process decreases hardness due to lamination process.

3.4. HC Fatigue strength

In Table 6-8, the staircase testing results are reported for the 3 investigated samples. In Table 9 the results processing: the stress amplitudes corresponding to 10, 50 or 90 % probability of failure within $2 \cdot 10^6$ cycles are here called $\sigma_{D\,(10\%)}$, $\sigma_{D\,(50\%)}$, and $\sigma_{D\,(90\%)}$.

Table 6. Staircase sequence for as received specimens.

Mean stress (MPa)	Alternate stress (MPa)	Cycles to failure
100	70	273’631
100	60	Run out
100	70	Run out
100	80	157’776
100	70	Run out
100	80	551’770
100	70	Run out
100	80	Run out
100	90	198’064
100	80	Run out
100	90	Run out
100	100	453’236
100	90	134’237
100	80	Run out

Table 7. Staircase sequence for 50°C treated specimens.

Mean stress (MPa)	Alternate stress (MPa)	Cycles to failure
100	100	106'682
100	90	318'208
100	80	438'839
100	70	348'045
100	60	Run out
100	70	Run out
100	80	Run out
100	90	394'265
100	80	301'352
100	70	1'437'006
100	60	1'203'321
100	50	Run out
100	60	Run out
100	70	564'346
100	60	Run out
100	70	Run out

Table 8. Staircase sequence for 80°C treated specimens.

Mean stress (MPa)	Alternate stress (MPa)	Cycles to failure
100	90	106'682
100	80	224'316
100	70	354'936
100	60	Run out
100	70	1'977'342
100	60	Run out
100	70	576'189
100	60	Run out
100	70	484'486
100	60	527'746
100	50	Run out
100	60	Run out
100	70	1'200'391
100	60	Run out

100	70	292'003
100	60	Run out
100	70	274'771

Table 9. Fatigue strength at $2 \cdot 10^6$ cycles. Staircase results for 10, 50 or 90 % failure probability and for as received, 50°C treated and 80°C treated specimens.

	as is	50°C	80°C
$\sigma_{D\,(50\%)}$	72	69	64
$\sigma_{D\,(10\%)}$	62	52	57
$\sigma_{D\,(90\%)}$	97	87	71

The value of $\sigma_{D\,(50\%)}$ decreases of 14% from as received to 80°C treated specimens. A similar trend is observed for $\sigma_{D\,(90\%)}$ and $\sigma_{D\,(10\%)}$. To interpret these data, some observations are required. Testing conditions are severe: 100 MPa tensile mean load is applied that is the crack opening is eased. The surface roughness parameters which follow this trend are *Rz* and *Rsm* that is the ones related to maximum distance between peaks and valleys and average distance between peaks. This means that with increasing process temperature the surface pits extend and become deeper. In Peter et al. 2019, the GE samples show an improved fatigue limit with respect to as received material, but the specimens mentioned in that reference were cast and not laminated. The investigated material is a B356.2 Aluminum alloy with lower mechanical properties (UTS 250 MPa e σ_y 144 MPa) and testing conditions less severe (R=0.1). The surface roughness of B356.2 specimens are worse than 7075 specimens.

In the present research, specimen axis is cut parallel to lamination direction. In as received specimens, roughness valley due to lamination do not cooperate with fatigue while, in GE processing, roughness is more homogeneous in direction 1 and 2. Furthermore, the effect on fatigue resistance of the increment in the depth of pits is more effective than the smoothing effect. If comparing this result with Peter et al. 2019, it can be observed that the GE process can be indicated for cast manufactured components.

3.5. SEM observation and EDS analysis

The SEM micrographs of the untreated and GE samples at different magnifications up to 1000x are reported in Figure 4. Scratches as well as some small (micrometric/submicrometric) holes are visible on the untreated sample (Figure 4 a-c). Appearance of the GE samples is different and it is characterized by micrometric cavities with rounded shape (Figure 4 (d)-(f)) homogeneously covering the surface, due to the etching process; some pits smaller in diameter and deeper in thickness can be evidenced. No substantial morphological differences can be evidenced between the samples etched at 50 and 80°C. EDS analyses of the samples are reported in Table 10.

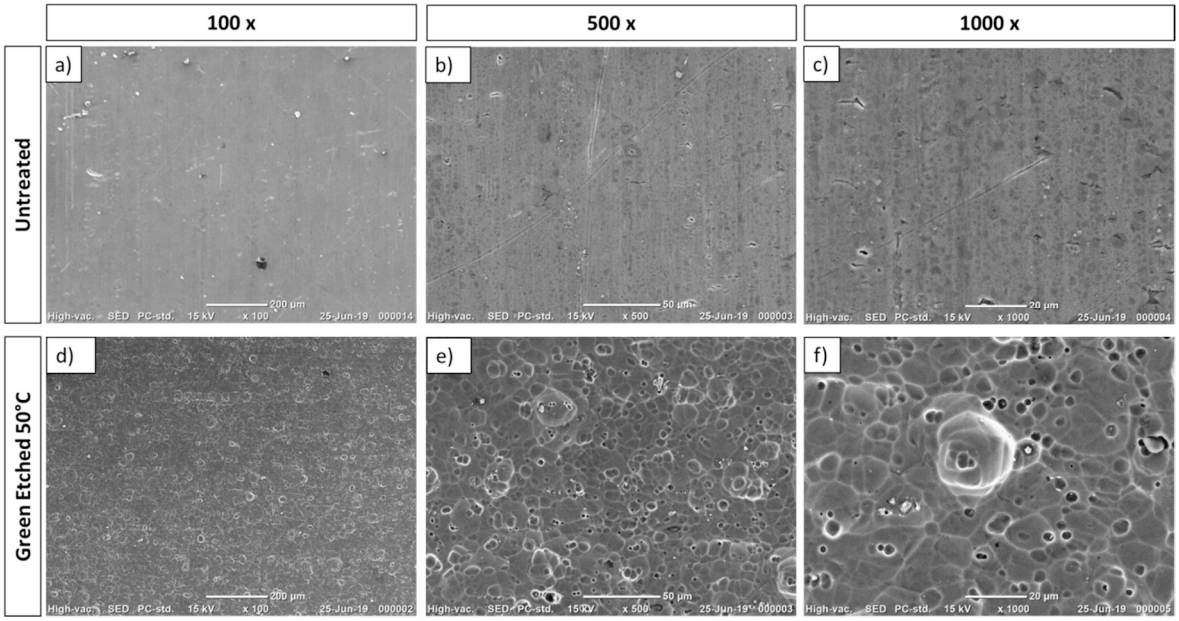


Figure 4: SEM images of untreated and GE samples.

Table 10: Chemical composition of the samples from EDS analysis

Element [%at]	Untreated	GE (50°C)
C	20.51	18.44
O	12.13	3.22
Na	0.56	
Mg	3.76	2.52
Al	61.02	73.03
Zn	2.02	2.80

No significant alteration in the surface chemical composition is observed after green etching, despite of a certain unexpected reduction in the oxygen content. This can be due to the fact that GE removed the pre-existent oxidated layer and new layer is thinner. For what concerns Al, Mg and Zn content, they are removed by GE and the removal is more effective for Mg than for Al and Zn.

3.6. Wettability

The values of contact angles are reported in Table 11. Green etching reduces surface wettability (increase in water contact angle). The difference between the contact angle of the untreated and of GE samples is statistically significant (p<0.05) while no significant differences can be evidenced between the two green etching conditions (p>0.05). The decrease in the surface wettability by water can be associated with the surface micrometric texture introduced by the green etching process, as observed at SEM.

Table 11: Contact angle values of untreated and etched samples

Sample	Contact angle [°]
Untreated	59 ± 10
GE (50°C)	79 ± 9
GE (80°C)	77 ± 6

3.7. Surface charge

Both the untreated and GE surfaces have a positive surface charge (around 100 mV) when in contact with a solution with pH in the range 5.5-8.5, but the GE surface shows higher standard deviation: this aspect is worth to be better explored because it could be related to higher surface reactivity as well as to higher surface electrical conductivity. On the other side, the GE surface maintains a positive surface charge with low standard deviation in the acidic range (pH 4-5.5) where the untreated surface is quite more reactive (or electrically conductive). These aspects could be investigated much more in details if surface reactivity is of interest for specific applications (corrosive environments, painting, joining or coating deposition).

4. Conclusions

The Green Etching® process is a chemical milling, obtained by means of dipping the component in alkaline solution. The process is cheap and sustainable: it requires low temperatures, the soak can be reused and recycled. It is safe for the operator.

Aim of the research is to investigate the effect of the Green Etching® chemical milling process on an aluminium alloy 7075-T6 manufactured by means of plastic deformation.
To this aim, preliminary measurements were performed on as received and 50°C and 80°C processed specimens: micrography, surface roughness, microhardness, SEM and EDS analysis, wettability and surface charge measurements.
Metallographic structure and microhardness did not result to be affected by the process as it is a surface treatment. Surface roughness showed to eliminate lamination traces and to generate pits which dimension increases with process effectiveness. Generally speaking, roughness increases with processing, but no relevant differences are observed with changing process parameters. Surface topography consists of micrometric rounded cavities after GE. Fatigue limit of the laminated material decreases with process temperature. This phenomenon appears to be more related to the height of the processing pits rather than to the smoothing effect on roughness. GE increases surface hydrophobicity and slightly changes surface charge and chemical reactivity in acidic environments.

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